

## A METHOD FOR OXYGEN DELIGNIFICATION OF CELLULOSE PULP AT HIGH PRESSURE IN SEVERAL STEPS

The present invention concerns a method for the improved oxygen delignification of cellulose pulp according to the introduction to claim 1.

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### The Prior Art

The first system with two-stage oxygen delignification was tested in Moss in Norway, and the first commercial two-stage system with remixing between the stages was subsequently implemented at Tomakomai mill in Japan, the

10 results of which were reported in the Tappi Proceedings Sept 8-10, 1992,

Japan-Tokyo, pp. 23-31. The principal aim of this remixing between the

stages, between the reactions, was to finely distribute residual chemicals in the pulp suspension and to break up any large gas bubbles to finely divided gas bubbles. Kvaerner Pulping (under its name at the time of "Kamyr AB")

15 took part in both of these systems, the one at Moss and the one at

Tomakomai, as supplier of MC-mixers and MC-pumps.

One of the first patents covering two-stage oxygen delignification is displayed in US 5,217,575, owned by Kvaerner Pulping AB, where the principles of

20 using an active heating before the second stage are reported. It is there

described in figures that improved delignification is obtained through

successively increased external heating between the stages or reactions, where the patent protects a more than approximately 20 degrees higher temperature in a second reactor. This, naturally, excludes the exothermic

25 heating that the oxygen reaction gives rise to of itself. Typically, an exothermic

reaction takes place, which gives a temperature increase of 5-8°C in the region of medium consistency, given an input kappa value of 25-30 units, which exothermic reaction is thus not included. It is specified in US 5,217,575

that a pressure of approximately 5 bar is established in the two reactors,

30 which creates, in practical application in such systems having only one pump

before the first reactor, a pressurisation of 5-8 bar in the first reactor and a pressurisation of 4-6 bar in the second reaction due to the large fall in pressure through the system that is caused by the viscously flowing pulp of

medium consistency. A fall in pressure of 0.05-0.1 bar per meter of pipe is typically developed in these systems. The second mixer between the reactors was primarily intended to provide a remixing of residual chemicals and thus not necessarily with the addition of further chemicals, where the second stage 5 becomes more of an extended alkali extraction with residual chemicals from the first stage.

A later system is revealed through, for example, the patent SE505141 (equivalent to US 6,221,206; US 6,319,357 and US 6,454,900) in which at 10 least 25 kg of alkali and 25 kg oxygen per tonne of pulp is to be initially added batchwise in a system with two reactors placed in series. Only one remixing takes place between the reactors, but this may take place with the addition of a small amount of alkali. A higher pressure is established in the first reactor in these systems with a highest specified pressure of 10 bar and a lower 15 pressure of a maximum of 5 bar in the second reactor. The same applicant, Sunds Defibrator, now named "Metso Paper", has demonstrated in the subsequent patent SE 510740 (equivalent to US 6238517) a second variant with 4-15 bar in the first reactor and 2-5 bar in the second reactor, and the applicant has described a third and a fourth variant in SE 507871 and 20 SE 507870 with 3-10 bar in a first reactor of upward flow and less than 2 bar in a second reactor of downward flow. It can be seen from this plethora of variants from the same applicant that the applicant has not realised the significance of the maintained high pressure in a second reactor following a remixing.

25 The maximal pressurisation of 12 bar follows the theories that have been adhered to by, for example, Metso Paper, and that are presented by Olm and Teder (Tappi Proceedings Seattle, 1979, pages 169-179), in which it is reported that no advantageous effects can be demonstrated at a higher 30 pressure than 10-12 bar. It must, however, be mentioned that these theories were established principally after laboratory experiments in which autoclaves were used in which only a small amount of pulp was bleached in a pulp specimen that was placed in the autoclave and rotated continuously or, laboratory mixers have been used that have contained a small amount of pulp

specimen under continuous stirring and often under continuous pressurisation with externally added oxygen (something that creates an unlimited access to oxygen).

- 5 Theories are presented in the patents WO97/17489 (STORA) and US 3725194 (SAPPI) concerning the positive effect of applying high pressure during the oxygen delignification with the aim of increasing the amount of oxygen that, at least initially, can be dissolved in the liquid phase. A pressure of 15-20 is applied in STORA's application, while SAPPI's patent describes
- 10 pressures of up to 400 psi (approximately 27 bar). Also these solutions attempt to achieve an improved delignification in which the fraction of oxygen that is dissolved in the fluid phase is high.

A specific sequence O-C/D-O-D is patented in another patent, SE 369746.

- 15 The first and the second oxygen stages in this sequence are carried out very aggressively at a pressure of 14 bar and temperatures of 119 °C and 130 °C, respectively.

A two-stage oxygen delignification is described also in EP 865531, where repeated mixing takes place between reactors. It is, however, specified in this case that the pressure lies within the interval from 20 psig to the maximum pressure of 180 psig (i.e. from 1.37 to 12 bar). The significance of repeated mixing at high pressure in order to optimise the fraction of dissolved oxygen has not been recognised at all here. A mixer followed by a reactor provided

20 with a stirrer is shown in a variant shown in Figure 4 of the patent, which stirrer can contribute to a repeated stirring effect in the reactor.

- 25 A process is suggested also in Camilla Rööst's thesis, *The Impact of Extended Oxygen Delignification on the Process Chemistry in Kraft Pulping*, ISSN 1652-2443, May 2004, with two oxygen reactors in which an optimisation has been attempted of the two-stage technology according to US 5,217,575 with heating between the stages, and in this way a powerful delignification system in which a pressure of 16 bar is established in the first reactor and a pressure of 6 bar has been established in the second reactor

has been seen. Pressures of 6, 10 and 16 bar in the first reactor have been tested during the optimisation, and it has proved to be the case that the pressure, together with the addition of alkali, is one of the most dominant parameters for good delignification.

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Other solutions have been presented with repeated remixing during oxygen delignification. A system with three mixers in series, in which a pressure of 120 psig (approximately 8.2 bar) is established, is described in US 5,460,696. The aim in this case is to reduce a pH that is far too high if all alkali is added 10 batchwise at the beginning, and the repeated mixing takes place with the aim of mixing alkali in gradually, as the alkali is consumed.

Three mixers in sequence are shown in US 4,384,920 and US 4,363,697, where these mixer stations are constituted by horizontal reactors with an 15 internal feed screw and stirring screw. The pulp is fed through the horizontal reactors while a gas phase is established at the roof of the reactors. These systems are clearly run at a moderate excess pressure.

A system is shown in US 4,259,150 in which the pulp is led directly from the 20 digester, while maintaining full digester pressure, through four mixers in series, for the addition of oxygen. In-line drainers are used in this case to drain off fluid with precipitated organic material, and this has the result that the concentration successively rises. This system, however, is very expensive since large quantities of oxygen are required since the pulp suspension on 25 exit from the digester contains a very large fraction of oxidisable organic material in the fluid phase, and significant amounts of fibre bundles for which the fibre removal process, the digestion process, is incomplete, and the bundles have a high content of lignin, accompany the pulp since no straining operation precedes the oxygen treatment, which bundles of fibres consume 30 large quantities of oxygen. It is here claimed that it is possible to adjust the digestion process such that the digested pulp obtains an increased kappa value around 70, rather than the normal value of 35, after which delignification can take place down to a kappa value of 15. An improved selectivity can be obtained in this manner, i.e. it is possible to reach the same kappa value but

with a higher pulp strength. This process is not one that has been driven to a great degree, since most of the oxygen delignification stages in the bleaching line act on cellulose pulp that has an input kappa value of this level.

- 5 There have been executed innumerable experiments in which it has been attempted to drive the delignification at medium consistency further than what it has become clear is possible to achieve in a mill environment. Verification of new processes often takes place in laboratories using autoclaves or laboratory mixers, which (in contrast to the continuous processes in a mill)
- 10 often takes place under continuous stirring of the pulp specimen and in certain cases with pressurisation by oxygen from an external source, which ensures an excess of oxygen during the complete process, and in the presence of saturated oxygen in all parts of the treated pulp specimen. It is often therefore possible to drive the oxygen delignification further in laboratory specimens
- 15 than in a mill environment.

The mixing of the chemical, oxygen and alkali, has been considered by some to be significant, and several solutions involve at least one of the establishment of a high pressure and the addition of surfactants in order to maintain the finely distributed gas phase evenly distributed throughout the

20 cellulose pulp during the process. Theories have been presented in which a maximal contact area is to be maintained between the gas phase and the fluid phase, something that can take place through minimising the size of the finely divided gas bubbles, such that the dissolving of the oxygen in the gas phase into the fluid phase can be promoted over a maximised transition surface. The

25 physical process in which the oxygen is dissolved proceeds, however, relatively slowly compared with the rate of consumption of oxygen during the first phases of the reaction, and it requires that the fluid phase that locally surrounds the oxygen bubble has a lower level of dissolved oxygen than is theoretically possible in the process conditions that are prevalent. The oxygen

30 is considered, however, to react primarily with the lignin in the fibre (after the oxidisable material in the fluid phase has reacted), and that the fluid phase that has penetrated the fibre is not immediately surrounded by oxygen bubbles, which ensures that the oxygen that is to react with the fibre material (and to reduce the kappa value) must first pass into solution from the gas

phase to the fluid phase, and then diffuse into the fibre in the fluid phase, and that all of this is to take place without the dissolved oxygen being consumed first by organic material in the fluid phase. This results in the fibre having a constant low value of dissolved oxygen with which it can react, something that  
5 is not advantageous for the process.

### Aim and Purpose of the Invention

The present invention intends to improve the oxygen delignification at medium consistency, in the region of pulp consistency 8-16%, hereafter referred to as  
10 "MC", in a mill environment such that delignification can be driven as far or further than what is possible when testing in laboratories. It is also possible with the invention to maximise in a mill environment the fraction of dissolved oxygen in the fluid phase and to promote the penetration of the oxygen-saturated fluid into the fibre if a very high pressure is maintained, and to  
15 subject the pulp to a repeated mixing at this high pressure.

The principles that are applied in the invention are the establishment in a first high pressure section of a pressure that is greater than the 12-15 bar that has been set in most commercial systems as the maximum suitable operating  
20 pressure, and the initiation at this higher pressure of remixing effects in the pulp suspension, with or without the extra addition of chemicals, with the aim of ensuring that the part of the oxygen that has been dissolved in the fluid phase is held as high as possible throughout the complete volume of the fluid and that the fibre is stirred in this volume of fluid that has been saturated with  
25 oxygen such that the fluid that has been saturated with oxygen can be allowed to penetrate the fibre in a more effective manner. The remixing effects are initiated according to the invention in a manner that is adapted to the decreasing rate of reaction in the delignification, such that these remixing operations take place after successively greater retention times in the high  
30 pressure section between the remixing operations.

It is typical that a pressure greater than 15-20 bar or higher is established in this high pressure section.

Other advantages and aims are made clear by the following description of embodiments.

### Description of Drawings

Figure 1 shows schematically a system for oxygen delignification in which the method according to the invention can be applied;

5       Figure 2 shows in principle how oxygen is mixed in to be mixed with the cellulose pulp as finely divided or as a dissolved fraction during the process as described by Figure 1;

10      Figure 3 shows how oxygen is consumed in an alternative system with three remixer positions during the high pressure phase and with maintained pressure, and with a mixing effect in the final reactor of the high pressure section;

15      Figure 4 shows how the oxygen is consumed in a further alternative system with only 14 kg of batchwise added oxygen and two remixing positions placed close to each other;

20      Figure 5 shows how oxygen is consumed in a two-reactor system with a high pressure zone and a low pressure zone without repeated remixing in the high pressure zone.

### Detailed Description of Preferred Embodiments

25      Figure 1 shows an oxygen delignification system between a preceding wash  $W_1$  and a subsequent wash  $W_2/W_3$ , with a number of reactors  $R_1, R_2, R_3$  and  $R_4$  between the washes.

The pulp is passed after the first wash  $W_1$  to an atmospheric storage tower ST (or to an atmospheric pulp chute).

30      It is appropriate that the alkali that must be added batchwise to the oxygen is added batchwise at the bottom of the storage tower ( $NaOH_{MAIN}$ ) after which all subsequent pumps (P1/P2) contribute to efficient mixing, since pumps are good mixers of fluid additions such as, for example, alkali ( $NaOH$ ).

The three first reactors  $R_1, R_2$ , and  $R_3$  are part of a high pressure section that establishes its pressure by two pumps P1 and P2 connected in series. The first pump P1 is a fluidising MC-pump with degassing, which not only is necessary in order to fluidise the MC-pulp such that it can be pumped, but also is used to separate out air (Air) from the pulp, which air otherwise would influence the process negatively from the point of view of delignification (not a

- high level of oxygen or a content of other residual gases) and reduce the opportunity of pressurising the pulp in an optimal manner. Also the second pump may be a fluidising MC-pump, but in this case without degassing, although it is appropriate that it is a conventional centrifugal pump optimised
- 5 for pressurisation, since the pulp has already been fluidised and degassed by the first pump. It is appropriate that the second pump has a significantly higher pumping efficiency, normally a pumping efficiency that lies 10-20% higher than that of the first pump.
- 10 The pumps P1, P2 establish initially in the high pressure section R<sub>1</sub> - R<sub>3</sub> a pressure that lies well over 15.0 bar, typically in the interval 17-30 bar and preferably in the interval 17-25 bar. The pressure may be higher, but it is limited by practical considerations to 17-20 bar with two pumps connected in series. If a pressure level of, for example, 18 bar is to be established after the pumps P1/P2, a suitable dimensioning and choice of pumps can be carried out such that the first fluidising MC-pump P1 establishes a pressure height of 6-8 bar (approximately 30-40%), and the subsequent centrifugal pump establishes a pressure height of 12-14 bar (approximately 60-70%), i.e. approximately one third of the total required pressure height is achieved in the
- 15 first MC-pump and the remaining pressure height in the second pump. If a higher pressure is required, several pumps can be connected in series, where also a third and final pump can be a centrifugal pump.
- 20 It is preferable that oxygen is added after the pressurisation by the pumps P1/P2 to the pressurised cellulose pulp through a principal mixer M1. It is appropriate that the mixer M1 should be a high intensity mixer with retention times of at least 0.1-2.0 seconds, which mixes oxygen evenly into the cellulose pulp in a powerful shear force field.
- 25 The pressurised pulp is led from the first principal mixer M1 at a pressure of approximately 18 bar with the oxygen that has been mixed into it to a first reactor R<sub>1</sub> in which the pulp flows upwards with a first retention time t<sub>1</sub> in the reactor R<sub>1</sub>.

After the pulp has passed through the first reactor R<sub>1</sub> the pulp is led to a second remixing location in the form of the mixer M2. This mixer may be of a simpler type than the principal mixer M1, and the principal aim is to obtain a remixing effect in order to increase the dissolved fraction of oxygen and to

5 promote the penetration of the saturated fluid phase into the cellulose fibre.

The fall in pressure across this mixer is to be kept as low as possible, preferably well under 1 bar, and the mixer may in its simplest form be a static mixer, possibly in the form of a half-closed valve. The mixer M2 may also be a small and simple pump (with a limited build-up of pressure that corresponds to

10 the fall in pressure in the pipes and system up to this point), or it may be an agitation and fluidising mixer.

The pressurised pulp is led from the second remixing location by the mixer M2 at a pressure of approximately 17 bar with its remixed oxygen to a second reactor R<sub>2</sub> in which the pulp flows upwards with a second retention time t<sub>2</sub> in

15 the reactor R<sub>2</sub>.

After the pulp has passed the second reactor R<sub>2</sub> the pulp is led to a third remixing position in the form of the mixer M3. This mixer may be of the same type as the mixer M2 with the same aim and the same fall in pressure.

20 The pressurised pulp is led from the third remixing location at a pressure of approximately 16 bar with its remixed oxygen to a third reactor R<sub>3</sub> in which the pulp flows upwards with a third retention time t<sub>3</sub> in the reactor R<sub>3</sub>.

25 The high pressure section ends after the third reactor R<sub>3</sub> and a controlled and managed reduction in pressure is thereafter carried out before the concluding low pressure section in at least one final reactor R<sub>4</sub>.

The reduction in pressure can be implemented through at least one valve, although it is preferable that several valves V<sub>1</sub>, V<sub>2</sub> and V<sub>3</sub> are used, where it is ensured that the fall in pressure is kept as low as possible across each valve

30 in order to avoid large and sudden falls in pressure that risk unnecessarily the flashing out of gas in the form of large bubbles. Several valves in series ensures that each valve also provides a stirring mixing effect in the turbulence that is formed, which partially counteracts the negative effects of the fall in

pressure by maintaining the oxygen evenly distributed in the form of small bubbles.

The low pressure section is, however, to continue to have a relatively high pressure in order to be able to maintain a finely distributed residual amount of 5 the oxygen in the cellulose pulp, but it is to have a sufficiently low pressure such that the pulp can be heated by the addition of direct steam(MP steam) from the medium pressure steam network of the mill. The medium pressure steam in these networks is normally held at a pressure of 10-12 bar, and a positive pressure difference of at least 1-2 bar is required in order to be able 10 to introduce steam into the pulp. The pressure difference required depends on the particular mixer M4, where this medium pressure steam is added to the pulp after the establishment of the correct pressure following the controlled pressure reduction after the valves V1-V3.

It is appropriate that a pressure less than 10-12 bar is established after the 15 high pressure section, the level being determined by the pressure in the steam network for medium pressure steam at the mill, and this pressure after the high pressure section should be at least 1-2 bar lower than the pressure in this steam network, with conventional mixing methods for the steam. If the pressure difference is lower, an arrangement must be used for the addition of 20 steam in which steam is added into the pulp through sluices without the risk of the pulp flowing out against the flow of steam.

The addition of steam entails the raising of the temperature of the cellulose 25 pulp by at least 5 °C followed by the leading of the heated pulp to a reactor system in a low pressure part, with a retention time  $t_4$  that exceeds the total retention time ( $t_1+t_2+t_3$ ) in the high pressure section.

The reactors  $R_1$ ,  $R_2$  and  $R_3$  in the high pressure section are so dimensioned 30 that the cellulose pulp is given successively longer retention times for the cellulose pulp, such that when the number of high pressure reactors is X, then the retention time for the reactors  $t_{1-X}$  is such that  $t_1 < t_2 < \dots < t_X$ , where  $t_1$  is the retention time in reactor  $R_1$ , etc. This ensures adaptation to the reaction process, in the form of consumption of the added chemicals, something that occurs very rapidly initially and subsequently falls essentially exponentially.

Suitable retention times in the reactors  $R_1$  to  $R_X$  in the high pressure section can be approximately expressed as:

$$t_{\min} = 1 \text{ minute for } t_1, \text{ after which } (t_x=2 * t_{x-1}) \text{ and } t_{\max}= X * 10 \text{ minutes}; \\ (t_1=1-10, t_2=2-20; t_3=4-30; t_4=8-40 \text{ min., etc}),$$

5 where  $t_x < t_{x+1}$ .

- With the aim of further improving the fraction of dissolved oxygen in the complete fluid volume in the high pressure section, a stirrer can be placed in at least one high pressure reactor that acts in the main part of the reactor
- 10 volume (it is to act in greater than 50% of the reactor volume), either in the form of a mechanical stirrer (S) or a hydrodynamic stirrer that at least circulates free fluid in the reactor. This stirrer may be present, for example, in the largest reactor in the high pressure section, or in the reaction in which the consumption is most rapid (which is the first reactor), or in all reactors. The
- 15 mechanical stirrer can be realised in the form of a rotating shaft with arms that extend radially in the reactor, which stirrer is driven at a rather moderate rate of revolution of 10-100 rpm and does not exert any fluidising effect on the cellulose pulp.
- 20 The retention time in the heated low pressure section is greater than the total time in the high pressure section. Given a total retention time of at least 3-10 minutes in the high pressure section, a suitable minimum retention time in the low pressure section is at least 30 minutes, which gives a total retention time of at least 40 minutes. The high pressure section for these minimum
- 25 conditions can consist of a first reactor with a retention time of at least one minute, remixing, followed by a second reactor with a retention time of at least 2 minutes and, after a further remixing, a final third reactor in the high pressure section with a retention time of at least 4 minutes.
- The low pressure section may have a retention time in the interval 30-120
- 30 minutes, preferably 60-90 minutes.

In a plant that delignifies 3,000 tonnes of MC-pulp at a consistency of 10% every day, the flow through the process is just over 18 cubic metres per minute. Pipes with a diameter of approximately 1.2 metres are normally used

for such a level of production, and these contain approximately 1.1 cubic metres per metre of pipe. A first reactor which has then be implemented in the form of a single pipe with a retention time of 1 minute will then be approximately 16 m long. It is often attempted to realise the reactors as pipes,  
5 which is the principal reason for keeping the retention times in the reactors low. A pipe of length 16 metres can be formed in the shape of a U-bend in a vertical plane, or it can be located in a horizontal plane.

The pressure in the cellulose pulp is released after the low pressure section at  
10 the outlet from the reactor R4 and the pulp is fed to an atmospheric pulp chute (or to a storage tower) that can lead off residual gases, after which the pulp from which the pressure has been released is pumped by pump P3 to the subsequent washing system, which is shown in Figure 1 in the form of a pressure diffuser W<sub>2</sub> and a washing press W<sub>3</sub> placed in series. This  
15 combination can become relevant if very high requirements are placed on the cleanliness of the pulp, for example, if the pulp is to be used as packaging for foodstuffs. It is, however, often sufficient with a single washing machine, and this reduces the investment costs. Washing filtrate F3 is led in a conventional manner from the final wash W<sub>3</sub> as washing fluid F2 for the next to last wash  
20 W<sub>2</sub>, i.e. the washing filtrate is led as a countercurrent flow relative to the flow of pulp. The washing filtrate F1 from the wash W<sub>2</sub> directly after the final reactor R<sub>4</sub> is led in an equivalent manner to a wash W<sub>1</sub> before the oxygen delignification as at least one of washing fluid and dilution fluid for the storage tower ST after this wash W<sub>1</sub>. The pulp that has been subject to oxygen  
25 delignification and washed is then pumped by the pump P5 to subsequent bleaching stages in the bleaching sequence.

Figure 2 shows in principle how oxygen is consumed in a system as portrayed in Figure 1, with the amount of oxygen plotted along the Y-axis and time plotted along the X-axis. The total batch of oxygen that is added in the mixer M1 is here 20 kg per tonne of pulp, which batch size lies in the upper region of the interval of 14-20 kg per tonne of pulp that is conventionally added batchwise to a oxygen delignification stage. It is not normally possible to add much more than 30-35 kg oxygen per tonne of pulp to a mixing-in position at a

pressure of 12 bar since such large quantities of gas readily give rise to the formation of channels in subsequent reactors, something that establishes a central flow through the reactor with very high speed, and a retention time in the reactor of only a fraction of the intended retention time. The high pressure  
5 that is applied according to the invention during the remixing and in the subsequent reactor results also in the ability to add greater amounts of gas without the risk of channels forming, or with a heavily reduced risk of channels forming, compared with systems having a lower pressure, given the same batchwise addition of oxygen. The residual quantity of oxygen that has not  
10 been consumed at any moment largely follows the line O<sub>2</sub> RES<sub>TOT</sub> and the consumption is initially very rapid. The symbols I, II and III denote the retention times in the reactors R<sub>1</sub>, R<sub>2</sub> and R<sub>3</sub>, respectively.

The maximum amount that can be dissolved in the fluid phase at the prevalent  
15 pressure is shown in Figure 2 by the grey-shaded region O<sub>2</sub> Liq<sub>MAX</sub>. At a pressure of 20 bar, at 80°C and at pH 10, approximately 0.5 kg of oxygen per cubic metre of fluid can be dissolved in the fluid phase. (The amount at a pressure of 10 bar is 0.25 kg). There is 9 cubic metres of fluid per tonne of pulp at a pulp consistency of 10%, and this means that a maximum of 4.5 kg  
20 of oxygen per tonne of pulp can be dissolved at a pressure of 20 bar. The amount 4.5 kg of a total batch size of 20 kg is equivalent to as much as 22.5% of the total batch size. The solubility of oxygen in the fluid phase is relatively low compared with those of many other bleaching agents, such as chlorine dioxide and other fluid-phase bleaching agents. The process in which oxygen  
25 passes from the gas phase to the fluid phase is relatively slow, but it can be accelerated through vigorous agitation in fluidising mixers. For this reason, thus, the first mixer M1 should either be a high-fluidising mixer with a relatively short retention time of 0.1-2.0 seconds, which vigorously agitates the mixture of pulp and gas in an intensive field of shear forces applied in a thin flow slit,  
30 or it should be a more moderate continuous agitation that occurs for a longer period of 1-10 seconds.

Given an optimal mixing in the mixer M1, it is possible to assume that the amount of oxygen that has been dissolved in the fluid phase lies close to the amount that theoretically can be dissolved in the fluid phase. The

consumption of that amount of oxygen that has been dissolved in the fluid phase, however, takes place very rapidly and considerably more rapidly than the transition of the oxygen from the gas phase to the fluid phase, and the fraction of remaining dissolved oxygen in the fluid phase follows the curve O<sub>2</sub> Liq RES for this reason. The oxygen that is to react with the fibre wall in the cellulose is, therefore, depleted far too rapidly, and this results in the oxygen delignification of the cellulose fibre occurring under conditions that are by no means advantageous. A remixing in the mixer M2 is for this reason activated, such that it is possible to increase the fraction of oxygen that is dissolved in the fluid phase. Also this dissolved quantity is consumed during the period II in the reactor R<sub>2</sub> and a further remixing with the mixer M3 for this reason takes place, in order again to increase the fraction of oxygen that is dissolved in the fluid phase. The oxygen that has been dissolved by the mixer M3 is subsequently consumed during the period III in reactor R<sub>3</sub>.

The system in Figure 1 is of conventional type, and the pressure thus falls through the system due to pressure loss in pipes, etc. The pressure falls for this reason in Figure 1 from reactor R<sub>1</sub> to R<sub>2</sub> by approximately 1 bar, and it falls from R<sub>2</sub> to R<sub>3</sub> with approximately 1 bar, exclusive of any pressure fall that depends on loss of static height. There will also be a pressure fall of 1 bar in a reactor with a height of 10 metres due to the difference in the static heights of the inlet and the outlet of the reactor. A high starting pressure of 17 bar thus gives approximately 16 bar in reactor R<sub>2</sub> and 15 bar in reactor R<sub>3</sub>. The fall in pressure through the high pressure section (HP) of the system (corresponding to the reactors R<sub>1</sub>, R<sub>2</sub>, R<sub>3</sub>) is to be minimised since the principle of the invention is to maintain the pressure at a high level as far as possible throughout the complete high pressure section HP, and to activate the remixing operations at a maintained pressure in order to maintain the fraction of oxygen that has been dissolved in the fluid phase as high as possible as far as it is practically possible.

The pressure in itself is important in order to facilitate the transition from gas phase to fluid phase since it is possible to maintain the area of contact between the gas phase and the fluid phase at a high level if undissolved oxygen (distributed in the suspension as either visible or invisible foam or

bubbles) can be maintained in the form of small bubbles and counteract the aggregation of these into large aggregates of gas, which happens if, among other effects, the pressure is reduced.

If the gas can be maintained in the form of bubbles with a diameter of 0.1 mm  
5 instead of 1 mm, the area of contact between the gas phase and the fluid phase can be increased by a factor of 100. It is also possible to maintain small bubbles distributed through the complete volume of the suspension, and they can penetrate fibre walls more easily.

10 The pressure is reduced after the high pressure section HP to a lower level in the low pressure section LP, to a pressure at which medium pressure steam can be used to heat the pulp suspension directly. At an appropriate pressure of 10 bar in the low pressure section, only half of the quantity of oxygen that can be dissolved at 20 bar can be held in solution. The remaining quantity of  
15 oxygen at this process position, however, is relatively low, due to the high consumption in the high pressure section. The amount of oxygen that theoretically can be dissolved at a pressure of 10 bar, however, does amount to 40-60% of the residual amount of oxygen that remains, and for this reason it may be the case in certain systems that it is desirable to add a small amount  
20 of further oxygen before the low pressure section.

In summary, Figure 2 shows how the fraction of oxygen dissolved in the fluid phase can be maintained at a high level by repeated remixing not only by remixing but also by retaining a high pressure. Both of these conditions are necessary in order to be able to dissolve a fraction as high as just over 20% of  
25 the total addition of oxygen and maintaining the amount of oxygen in the fluid phase at a high level throughout the complete high pressure section.

Advantageous conditions for delignification of the fibre wall at its contact with the fluid phase, which also penetrates into and between the fibres, are in this way created.

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Figure 3 shows another variant in which three remixing positions between four reactors are used, and a stirrer S is also present in the final reactor, as is shown schematically in Figure 1. The same high pressure throughout the complete high pressure section HP is established in this case, and this may

- be ensured where required by mixers at the remixing positions that raise the pressure. The mixers DUALOMIX™ from Kvaerner Pulping AB are mixers of a type that can give a build-up of pressure in certain applications (determined by the size and the flow) and with certain designs of the mixer. It is possible as
- 5 an alternative to insert auxiliary pumps between the reactors. In this case, again, 20 kg of oxygen has been added as a batch at the beginning, but the remixing operations take place more frequently with shorter and successively increasing retention times between the remixing positions.
- 10 Figure 4 shows a variant with an initial batchwise addition of oxygen of 14 kg, maintained pressure throughout the system and more frequent remixing operations than in the variant shown in Figure 2. Otherwise this variant is the same as that shown in Figure 2.
- 15 Figure 5 shows how the oxygen would be consumed if the method according to the invention were to be not applied in a two-reactor system with a first high pressure section and a second low pressure section, i.e. if remixing does not take place in the high pressure section. The drawing makes it clear that the amount of oxygen dissolved in the fluid phase falls rapidly to a very low
- 20 amount, and delignification of the fibre and the fibre wall takes place for this reason in conditions that are far from ideal in the latter part of the high pressure section.
- The main part of the chemicals, both oxygen and alkali, required is, in principle, to be added before the first reactor, which quantities of chemicals
- 25 are such that alkali is added to obtain an initial pH that lies well over 9.0 and oxygen is added at an amount of between 5 and 50 kg per tonne of pulp. The particular amount added depends on the initial kappa value. When using pulp with an initial kappa value of 40-50 units and with a reduction to a kappa value of 8-10, the addition of oxygen can amount to 30-50 kg per tonne of pulp, i.e.
- 30 an effect on the kappa value of 1-2 kg per  $\Delta$ kappa and tonne of pulp. If the initial kappa value is lower, the kappa factor typically lies between 1.5 and 3.0 kg per  $\Delta$ kappa and tonne of pulp. Thus the invention is applied in an oxygen

delignification with a kappa factor addition of oxygen that lies in the region 1.0-3.0 (kg oxygen per  $\Delta$ kappa and tonne of pulp).

Alkali must be added such that the pH is maintained at a final value of 10-10.5 such that the alkalinity is maintained at a sufficiently high level during the 5 complete process. This normally means that 80-100% of the total amount of oxygen is added at the first mixing-in position, while 70-90% of the total amount of alkali is added at the same mixing-in position. In certain cases, in particular primarily in cases in which the total retention time is short, the complete amount of alkali can be added at this position.

10 Normally, only alkali is added before the low pressure section principally for long total retention times, together with a small amount of oxygen if the retention time in the low pressure section is long. It has proved to be the case in practice that the vast majority of systems display improved delignification and an improved strength of the pulp, i.e. an improved selectivity, if a small 15 amount (typically 10%-40% of the total amount) is added as a batch at the second stage.

Adaptation of the strategy for additions takes place depending on a number of factors, such as:

- 20 • the initial kappa value, where a higher initial kappa value may entail a greater number of addition points for alkali or oxygen;
- the total kappa reduction during the oxygen delignification;
- the current cellulose being used (deciduous wood, conifer wood, heavy eucalyptus, etc., where short-fibred deciduous wood may require more frequent or a greater number of remixing positions in the high pressure 25 section);
- the subsequent bleaching stages (the ECF or the TCF sequence, the use of other alkali bleaching stages of P-stage type, and the power and the number of alkali extraction stages of E-/EO-/EOP-type);
- 30 • the properties desired for the bleached pulp;
- requirements on the COD level of emissions (this may require more severe conditions in the oxygen treatment despite reduced selectivity, which may reduce the amount of chlorine dioxide required in D-stages).

With the aim of obtaining an optimal effect on the cellulose pulp, and of not consuming the chemicals on unnecessary organic material in the fluid phase, the cellulose pulp may be dewatered to give a higher consistency before the 5 oxygen delignification and it may be rediluted to a medium consistency before the oxygen delignification using pure filtrate (filtrate that is obtained from washing stages after the oxygen delignification or other clean process water), which has preferably been oxidised before this in an oxidising reactor Ox.R. The alkali that is added may, for the same reason, be added in portions or 10 totally in the form of oxidised white liquor.

The invention may be varied in a number of ways within the framework of the invention. For example, types of reactor other than a tower of upward flow may be used, such as a tower of downward flow, or simple pipes that have 15 been laid in a horizontal plane or in a U-bend in a vertical plane.

Batchwise addition of oxygen may be required at all mixing positions in the system for certain pulps, such as deciduous wood pulps, that are difficult to delignify. The use of more than three reactors with preceding mixing 20 operations for each in the high pressure section may also be required, as may the use of more than one reactor in the subsequent low pressure section. However, at least one remixing position is to be established in the high pressure section with a predetermined minimum time delay following a preceding principal mixing position, together with a subsequent low pressure 25 section having at least one reactor.

The predetermined time delay between the mixing positions may be adapted to the current consumption of oxygen dissolved in the fluid phase or to the total amount of added oxygen, where it is appropriate that a remixing operation can take place as soon as possible after more than 30% of the 30 oxygen that has been previously added has been consumed. The preferred retention times for the pulp in the reactors R1-R3 that have been specified are, however, useful guidelines for the vast majority of processes, which guidelines have been adapted to the rapid consumption at the beginning of the process, a rate of consumption that gradually declines.

- The invention may also be applied in a delignification system with a short initial low pressure section in which a small amount of oxygen, typically considerably less than 40% and preferably less than 20% of the total that is added to the oxygen stage, is added before or at the low pressure section with
- 5 the aim of oxidising the material that is present dissolved in the fluid phase. It is possible in this manner to avoid unnecessary consumption of the oxygen that is added at the subsequent high pressure section by oxidisable material in the fluid phase, and ensure a greater fraction being used for delignification of the cellulose in the high pressure section.
- 10 It is preferable that also oxidised white liquor is used as addition of alkali.

Oxygen delignification in a mill environment can be more readily driven to lower kappa values with the method according to the invention, and deciduous wood, for example, which in laboratory trials can be delignified down to a

15 kappa value of 9, or lower, can approach this potential reduction in kappa also in a mill environment. The reduction in kappa value in a mill environment can, in certain conditions, be improved by up to 3 kappa units while retaining the strength of the pulp. It is alternatively possible to obtain the same reduction in kappa value with a considerably improved pulp strength, or intermediate

20 variants between these extreme alternatives may be achieved in which both the strength of the pulp and the reduction in kappa value are improved. The costs for the operation of the bleaching line, in particular the costs for further bleaching agents in subsequent bleaching stages, are considerably reduced if it is possible to reduce the kappa value by a further 2-3 units in the oxygen

25 delignification at an early stage of the bleaching line.